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Integrated Data Collection Analysis (IDCA) Program —$KClO_3$/Dodecane Mixture

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of a mixture of $KClO_3$ and dodecane—$KClO_3$/dodecane mixture. This material was selected because of the challenge of performing SSST testing of a mixture of solid and liquid materials. The mixture was found to: 1) be more sensitive to impact than RDX, and PETN, 2) less sensitive to friction than PETN, and 3) less sensitive to spark than RDX. The thermal analysis showed little or no exothermic features suggesting that the dodecane volatilized at low temperatures. A prominent endothermic feature was observed assigned to melting of $KClO_3$.

This effort, funded by the Department of Homeland Security (DHS), ultimately will put the issues of safe handling of these materials in perspective with standard military explosives. The study is adding SSST testing results for a broad suite of different HMEs to the literature. Ultimately the study has the potential to suggest new guidelines and methods and possibly establish the SSST testing accuracies needed to develop safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. The results are compared among the laboratories and then compared to historical data from various sources. The testing performers involved for the $KClO_3$/dodecane mixture are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and Indian Head Division, Naval Surface Warfare Center, (NSWC IHD). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to understand how to compare results when these things cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX, potassium chlorate, sugar, dodecane.

IDCA Explosives Safety Testing of Homemade Explosives
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1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives[1]. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture precursors are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—as well as taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

The IDCA has chosen several formulations to test that present these challenges. Table 1 shows the materials selected for the Proficiency Test and the Description column describes the form of the resulting mixture.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane[1]	Wet powder
Potassium chlorate	Dodecane[1]	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Ammonium nitrate		Powder
Bullseye® smokeless powder[4]		Powder
Ammonium nitrate	Bullseye® smokeless powder[4]	Powder mixture
Urea nitrate	Aluminum	Powder mixture
Urea nitrate	Aluminum, sulfur	Powder mixture
Hydrogen peroxide 70%	Cumin	Viscous paste
Hydrogen peroxide 90%	Nitromethane	Miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	Sticky paste
Hydrogen peroxide 70%	Glycerine	Miscible liquid
HMX Grade B		Powder
RDX Type II Class 5		Powder (standard)
PETN Class 4		Powder (standard)

1. Simulates diesel fuel; 2. Contains 3 wt. % cornstarch; 3. Sieved to pass 100 mesh; 4. Alliant Bullseye® smokeless pistol gunpowder;

Evaluation of the results of SSST testing of unknown materials, such as the HMEs in Table 1, is generally done as a relative process, where a well understood standard is tested alongside the HME. In many cases, the standard employed is PETN or RDX. The standard is obtained in a high purity, narrow particle size range, and measured frequently. The performance of the standard is well documented on the same equipment (at the testing laboratory), and is used as the benchmark. The sensitivity to external stimuli and reactivity of the HME (or any energetic material) are then evaluated relative to the standard.

Most of the results from SSST testing of HMEs are not analyzed any further than this. The results are then considered in-house. This approach has worked very well for military explosives and has been a validated method for developing safe handling practices. However, there has never been a validation of this method for HMEs. Although it is generally recognized that these SSST practices are acceptable for HME testing, it must always be kept in mind that HMEs have different compositional qualities and reactivities than conventional military explosives.

The IDCA is attempting to evaluate SSST testing methods as applied to HMEs. In addition, the IDCA is attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining the HMEs. The IDCA team has taken several steps to make this inter-laboratory data comparison easier to analyze. Each participating laboratory uses materials from the same batches and follows the same procedures for synthesis, formulation, and preparation. In addition, although the Proficiency test allows for laboratory-to-laboratory testing differences, efforts have been made to align the SSST testing equipment configurations and procedures to be as similar as possible, without significantly compromising the standard conditions under which each laboratory routinely conducts their testing.

The first and basic step in the Proficiency test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials are being tested in triplicate and RDX will continue to be tested throughout the IDCA Proficiency test.

The subject of this report, $KClO_3$/dodecane mixture, is the third in a series of materials that fall in the class of solid oxidizer/fuel mixtures and the first that is a mixture of solid oxidizer and liquid fuel. These materials were chosen for study in the Proficiency Test because of the challenge of testing a fine solid mixed with a non-viscous liquid fuel—adequate mixing on a small scale, representative sampling of a physical mixture, and handling a component that is volatile. The solid was dried as previously described[2] and separated through a 40-mesh sieve.[3] The dodecane was used as received from the manufacture.

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and Indian Head Division, Naval Surface Warfare Center, (NSWC IHD).

2 EXPERIMENTAL

General information. All samples were prepared according to the IDCA Program procedures for drying and mixing[2,4]. The $KClO_3$ was obtained from Columbus Chemical as a purified powder, Catalog #423000, Lot # 200917615, CAS # 3811-04-9, assay (by manufacturer): $KClO_3$, 99.7%; KCl, 0.05%; H_2O, 0.005%. The dodecane was purchased from Alfa-Aesar as *n*-Dodecane (99+%); Lot # L29T050 1 L, CAS # 112-40-3. The $KClO_3$ was dried for 16 h and cooled in a desiccator[2]. The $KClO_3$ was separated through a 40-mesh (425 μm hole size) sieve. The mixture was prepared by hand, adding the dodecane to the $KClO_3$ while stirring with a spatula in a materials compatible polypropylene container[4]. The mixture composition is 89-wt. % $KClO_3$ and 11-wt. % dodecane. The final mixture had the appearance of a wetted solid, with no evidence of free liquid in the vial. Typically, the precursors are mixed at that ratio to give approximately a 1-gram sample. This sample is divided up for the various SSST testing. Three samples were prepared this way and tested separately. The mixing ratio was selected to be stoichiometric[1], chosen for oxygen balance. The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for KC/Dodecane (89/11) Mixture [revised 3.31.11] (LLNL)[5], 50188 D KC/dodecane, revised 4.6.11 (LANL)[6], and KC/Dodecane (IHD)[7]. The sandpaper images were obtained on a JEOL 8400 Model number JSM-840A Scanning Electron Microscope operated at an accelerating voltage of 15 KV. The sandpaper samples were prepared by applying a thin coating of gold (~ 20 nm) to reduce surface charging.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the $KClO_3$/dodecane mixture.

Table 2. Summary of conditions for the analysis of $KClO_3$/dodecane mixture (All = LANL, LLNL, IHD)

Impact Testing

1. Sample size—LLNL and IHD, 35 ± 2 mg; LANL 40 ± 2 mg
2. Preparation of samples—All, dried per IDCA procedures[2]
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—All, Type 12*
6. Sandpaper—LANL, 150 grit or 180 garnet; IHD, 180 garnet; LLNL, 120 flint S/C or 180 garnet
7. Sandpaper size—LLNL, 1 inch square; LANL, 1.25 inch diameter disk dimpled; IHD not specified
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL and IHD, 2.5 kg; LANL, 0.8 kg
10. Positive detection—LANL and LLNL, microphones with electronic interpretation as well as observation; IHD use observation
11. Data analysis—All, modified Bruceton and TIL; LANL Neyer also

Friction analysis

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures[2]
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL and LLNL, BAM; IHD, BAM and ABL
6. Positive detection—All, by observation
7. Room Lights—LANL on; LLNL off; IHD, BAM on, ABL off
8. Data analysis—All, modified Bruceton and TIL

ESD

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures[2]
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL and IHD, ABL; LLNL, custom built*
7. Positive detection—All, by observation
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry

1. Sample size—All, ~ <1 mg
2. Preparation of samples—All, dried per IDCA procedures[2]
3. Sample holder—All, hermetic with pin hole; LLNL also uses sealed pan
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C
6. Pan hole size—LLNL, 50 μm; LANL and IHD, 75 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000*

Footnotes: *Test apparatus, *Impact:* LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LANL, IHD, AFRL —ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry:* LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

3 RESULTS

3.1 $KClO_3$/dodecane mixture

In this proficiency test, all testing participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in the IDCA report on method comparisons[8], which compares the different procedures by each testing category. LANL, LLNL, and IHD participated in this part of the SSST testing of the $KClO_3$. Screening the $KClO_3$ at -40 mesh was performed because the material seemed to naturally breakdown to a powder about this size with slight mechanical agitation. Because the composition of diesel fuel changes regionally and seasonally, dodecane was selected as a surrogate. Although $KClO_3$ and dodecane mixtures can be made at a variety of mixing ratios, the ratio for this study was selected that conforms to stoichiometry.

3.2 Impact testing results for $KClO_3$/dodecane mixture

Table 3 shows the results of impact testing of the $KClO_3$/dodecane mixture as performed by LANL, LLNL, and IHD. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive test. LANL used both 150- and 180-grit sandpaper, IHD used 180-grit sandpaper, and LLNL used 120-grit flint paper and 180-grit sandpaper for the impact testing. All participants performed data analysis by normal modified Bruceton method[9,10] and LANL also performed data analysis by the Neyer method[11].

Table 3. Impact testing results for $KClO_3$/dodecane mixture

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	3/25/10	23.9	23	38.2	3.61	0.041
LLNL (120)	3/30/10	23.9	22	40.5	1.87	0.020
LLNL (120)	4/09/10	22.2	16	36.7	8.09	0.095
LLNL (180)	5/13/11	23.3	20	9.0	1.12	0.054
LLNL (180)	5/16/11	23.3	18	9.6	1.06	0.048
LANL (150)	3/22/10	24.0	<10	12.6	4.74	0.160
LANL (150)	3/23/10	23.3	<10	9.0	3.24	0.153
LANL (150)	3/24/10	24.0	<10	12.1	1.51	0.054
LANL (180)	4/28/10	22.7	<10	6.4	0.90	0.061
LANL (180)	4/29/10	21.3	<10	7.6	0.47	0.027
LANL (180)	5/4/10	21.6	<10	10.2	1.89	0.080
IHD (180)	9/8/10	20	42	9	2.09	0.10
IHD (180)	8/24/10	20	45	12	1.94	0.07
IHD (180)	8/24/10	20	46	10	3.03	0.13

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. DH_{50}, in cm, by Modified Bruceton method, load for 50% reaction; 4. Standard deviation.

The test results from the three participating laboratories for impact show a large range for DH_{50} from 6.4 to 40.5 cm. The average values are LLNL, 26.8 ± 16.0 cm; LANL, 9.7 ± 2.5 cm; IHD, 10.3 ± 1.5 cm. The average values based on grit size are 120, 38.5 ± 1.9 cm; 150, 11.2 ± 2.0 cm; 180, 9.2 ± 1.7 cm. The standard deviation is below the 0.1 log unit range except for IHD, where one value is over 0.1 log units. This appears as a result of IHD using 0.1 log spaced steps while LANL and LLNL use 0.05 log spaced steps. The impact of step spacing will be evaluated in detail in a later report.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[11]. The DH_{50} values are in the same range as the values analyzed by the Bruceton method, where the averages for the Neyer method are 12.0 ± 1.7 cm and 7.9 ± 1.2 cm for the tests that used 150-grit and 180-grit sandpaper, respectively.

This trend is qualitatively similar to the 150-grit and 180-grit sandpaper, respectively in Table 3, when considering LANL data only. The Neyer analysis seems to accentuate the differences between the data sets from the two sandpapers.

Table 4. Impact testing results for $KClO_3$/dodecane mixture (Neyer or D-Optimal Method) 150- and 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (150)	3/22/10	24.0	<10	12.3	2.34	0.082
LANL (150)	3/23/10	24.0	<10	10.2	4.22	0.175
LANL (150)	3/24/10	23.0	<10	13.6	3.52	0.111
LANL (180)	4/28/10	22.0	<10	6.9	0.64	0.038
LANL (180)	4/29/10	22.0	15.4	7.6	0.51	0.029
LANL (180)	5/4/10	21.3	<10	9.3	1.53	0.071

1. Number in parentheses indicates grit size of sandpaper; 2 Relative humidity; 3. DH_{50}, in cm, is the Neyer method, load for 50% reaction; 4. Standard deviation.

3.3 Friction testing results for $KClO_3$/dodecane mixture

Table 5 shows the BAM Friction testing performed by LANL, LLNL and IHD. The difference in testing procedures by the three laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. All participants performed data analysis using the threshold initiation level method (TIL)[12], and a modified Bruceton method[9,10]. The average friction values for F_{50} are: LLNL, 25.5 ± 3.5 kg; LANL, 19.1 kg; IHD, 26.8 ± 3.3 kg. The standard deviation values range for all 0.03 < s < 0.408 cm, log units. The threshold values are in the following order LANL < LLNL < IHD. For 0 positive events, LANL and IHD recorded the similar values while LLNL have higher values.

Table 5. BAM Friction Testing results for $KClO_3$/dodecane mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, cm[5]	s, log unit[5]
LLNL	3/25/10	23.9	19	0/10 @ 11.2	1/10 @ 12.0	28.4	1.70	0.026
LLNL	3/31/10	22.2	18	0/10 @ 12.8	1/10 @ 14.4	21.6	15.15	0.284
LLNL	4/08/10	23.9	19	0/10 @ 12.8	1/10 @ 13.6	26.4	4.95	0.081
LANL	3/22/10	24.0	<10	NA[6]	NA[6]	>20.2[7]	NA	NA[7]
LANL	3/23/10	24.0	<10	NA[6]	NA[6]	>20.4[7]	NA	NA[7]
LANL	3/24/10	22.7	<10	NA[6]	NA[6]	19.1	5.34	0.12
LANL	3/22/10	23.6	<10	0/10 @ 7.2	1/3 @ 9.6	NA[8]	NA[8]	NA[8]
LANL	3/23/10	24.0	<10	0/10 @ 7.2	1/6 @ 9.6	NA[8]	NA[8]	NA[8]
LANL	3/24/10	23.0	<10	0/10 @ 7.2	1/2 @ 9.6	NA[8]	NA[8]	NA[8]
IHD	8/25/10	25	41	0/10 @ 14.7	1/7 @ 16.3	NA[8]	NA[8]	NA[8]
IHD	8/25/10	25	41	0/10 @ 18.4	1/5 @ 19.6	NA[8]	NA[8]	NA[8]
IHD	8/25/10	35	42	0/10 @ 16.3	1/4 @ 18.4	NA[8]	NA[8]	NA[8]
IHD	9/8/10	26	41	NA[6]	NA[6]	30.3	32.84	0.408
IHD	9/8/10	26	41	NA[6]	NA[6]	23.7	25.69	0.408
IHD	9/8/10	26	41	NA[6]	NA[6]	26.4	18.79	0.290

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. F_{50}, in kg, Modified Bruceton method, load for 50% Reaction, LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. Not applicable, separate sample used for TIL analysis; 7. BAM spacing for LANL became larger than 2.4 kg above the 24 kg level, invalidating the full Bruceton analysis for this sample; 8. Not applicable, separate sample used for Bruceton analysis.

Table 6 shows the ABL Friction testing performed by IHD on the $KClO_3$/dodecane mixture. IHD was the only participant to report ABL Friction testing results. LANL did not have the system in routine performance at the time. LLNL does not have ABL Friction. The results show the F_{50} is about 498 ± 20 psig @ 8 fps and the threshold 135 psig @ 8 fps.

Table 6. ABL Friction testing results for $KClO_3$/dodecane mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[2,4]	F_{50}, psig/fps[2,5]	s, psig/fps[6]	s, log unit[6]
IHD	8/26/10	25	40	0/20 @ 135/8	1/4 @ 180/8	479/8	180/8	0.16
IHD	8/26/10	25	41	0/20 @ 135/8	1/1 @ 180/8	518/8	315/8	0.25
IHD	8/26/10	25	41	0/20 @ 135/8	1/3 @ 180/8	497/8	163/8	0.14

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 4. Next level where positive initiation is detected; 5. F_{50}, in psig, Modified Bruceton method, load for 50% Reaction; 6. Standard deviation.

3.4 Electrostatic discharge testing of $KClO_3$/dodecane mixture

Electrostatic Discharge (ESD) testing of the $KClO_3$/dodecane mixture was performed by LANL, IHD and LLNL. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape and what covers the sample. In addition, LLNL uses a custom built ESD system with a 510-Ω resistor in series to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. (LLNL has purchased a new ABL spark tester and is being used for the spark testing on the 3rd RDX calibration run and the remaining IDCA threats.) All participants performed data analysis using the threshold initiation level method (TIL)[12].

Table 7. Electrostatic discharge testing $KClO_3$/dodecane mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	3/25/10	23.9	21	0/10 @ 1.0[5]	0/10 @ 1.0[5]
LLNL[4]	3/30/10	23.9	22	0/10 @ 1.0[5]	0/10 @ 1.0[5]
LLNL[4]	4/8/10	23.3	22	0/10 @ 1.0[5]	0/10 @ 1.0[5]
LANL	3/22/10	24.0	<10	0/20 @ 0.125	2/10 @ 0.25
LANL	3/23/10	24.0	<10	0/20 @ 0.125	2/2 @ 0.25
LANL	3/24/10	22.7	<10	0/20 @ 0.125	2/2 @ 0.25
IHD	8/24/10	23	40	0/20 @ 0.165	1/4 @ 0.326
IHD	8/24/10	23	40	0/20 @ 0.095	1/11 @ 0.165
IHD	8/24/10	23	40	0/20 @ 0.165	1/1 @ 0.326

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. LLNL uses a 510-Ω resistor in the discharge unit to mimic the human body.

LANL and IHD testing results find about the ESD sensitivity for the $KClO_3$/dodecane mixture. The data from LLNL show a non-sensitive material. This is expected because of the experimental configuration.

3.5 Thermal testing (DSC) of $KClO_3$/dodecane mixture

Differential Scanning Calorimetry (DSC) was performed on the $KClO_3$/dodecane mixture by LLNL, LANL, and IHD. All participating laboratories used different versions of the DSC by TA Instruments.

Table 8 shows the DSC data from each of the participating laboratories. For all three participants there is observed a sharp, high temperature endothermic feature with T_{min} values ranging from 357.5 to 359.5 °C. This is assigned to the $KClO_3$ melting from previous work on the thermal behavior of $KClO_3$/fuel mixes by TGA, DTA, and DSC[3,13,14]. IHD also observes a very small exothermic feature around 160°C.

Table 8. Differential Scanning Calorimetry results for $KClO_3$/dodecane mixture (pinhole hermetic pan), 10°C/min heating rate

Lab	Test Date	Endothermic, onset/minimum, °C (ΔH/J/g)	Endothermic, onset/minimum °C (ΔH, J/g)
LLNL	3/24/10	ND[1]	359.1/357.6 (161)
LLNL	3/24/10	ND[1]	358.0/359.2 (154)
LLNL	3/25/10	ND[1]	357.8/359.5 (159)
LANL	3/22/10	ND[1]	355.8/358.0 (148)
LANL	3/23/10	ND[1]	357.7/358.8 (142)
LANL	3/25/10	ND[1]	358.7/359.1 (182)
IHD	3/2/10	150.1/167.5 (12)	357.8/358.5 (168)
IHD	3/2/10	150.3/168.1 (8)	357/8/358.4 (129)
IHD	3/2/10	106.8/127.9 (24)	358.5/358.8 (123)

1. Not observed in this set of data.

Table 9 shows the DSC data, by LLNL, for the $KClO_3$/dodecane mixture where the DSC pan is closed instead of pinhole vented as used in the measurements shown in Table 8. The behavior of the profiles is substantially different than the behavior of the profiles for the pinhole vented samples. The hermetically sealed samples exhibit several exothermic events, including broad high temperature features. These high temperature features have been assigned by TGA and DTA as the decomposition of $KClO_3$. In addition, the endothermic event assigned to $KClO_3$ melting is also observed at ~ 350°C. The ΔH values of the lower temperature exothermic events add to about 400 J/g. The nature of the differences between Table 8 and Table 9 are discussed below.

Table 9. Differential Scanning Calorimetry results for $KClO_3$/dodecane mixture (closed hermetic pan), 10°C/min heating rate

Lab	Test Date	onset/minimum or maximum, °C (ΔH, J/g)
LLNL	3/24/10	Exo—202.9/211.2 (16); 230.7260.5 (207); 391.1 (175); 467.2; Endo—341.5; 352.7
LLNL	3/24/10	Exo—200.1/211.2 (24); 234.8/262.7 (184); 339.1 (175); 469.8; Endo—341.8; 355
LLNL	02/24/10	Exo—201.8/214.7 (14); 240.2/272.8 (223); 338.7 (184); 474.9; Endo—341.5; 352.7

4 DISCUSSION

Table 10 shows the average values for the data from each participant and compares it to corresponding data for standards, RDX and PETN. The data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test[15]. The data for PETN was provided by the participating laboratories (when available) from measurements performed outside this Proficiency Test. Table 10 allows the comparison of the average results on $KClO_3$/dodecane mixture with standards to obtain relative sensitivities.

4.1 Sensitivity of $KClO_3$/dodecane mixture compared to standards

Impact sensitivity. Table 3 shows the impact data where the testing was done using several different sandpapers. As a result, the combined data from all the laboratories covers a wide range of values. For the purpose of comparison, only the data from the testing with 180-grit sandpaper is used in Table 10. The effect of the grit size on the impact testing is discussed below in a separate section. The impact sensitivity varies little among the participating laboratories for the $KClO_3$/dodecane mixture, and the overall trend is that it is more impact sensitive than RDX as well as PETN.

Friction sensitivity. Although LANL results for BAM friction do not agree with LLNL and IHD results, when compared to the RDX standard, the F_{50} friction values for $KClO_3$/dodecane mixture are similar to RDX indicating it is about as sensitive to friction. When compared to PETN, the F_{50} friction values as well as TIL values indicate that the $KClO_3$/dodecane mixture is less sensitive.

Table 10. Average Comparison values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
$KClO_3$/dodecane[2]	9.3[3,4]	8.1[3,5]	10[3,5]	ND[6]
RDX Class 5 Type II[7]	24.1[8]	25.4[9]	19[3]	15.3[3]
PETN[10]	15	14.7	ND[5]	ND[6]
BAM Friction Testing[11,12]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
$KClO_3$/dodecane[13]	12.3[14]; 25.5[14]	7.2[14]; 19.1[15]	16.5[14]; 26.8[14]	ND[6]; ND[6]
RDX Class 5 Type II[7]	19.2; 25.1	19.2; 20.8	15.5; ND[6]	ND[6]; ND[6]
PETN[10]	6.4; 10.5	ND[6]; 9.2	ND[6]; ND[6]	ND[6]; ND[6]
ABL Friction Testing[16-19]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
$KClO_3$/dodecane[20]	ND[6]; ND[6]	ND[6]; ND[6]	135[21]; 498[21]	ND[6]; ND[6]
RDX Class 5 Type II[7]	ND[6]; ND[6]	ND[6]; ND[6]	74; 154	93; ND[6]
PETN[10]	ND[6]; ND[6]	ND[6]; ND[6]	ND[6]; ND[6]	ND[6]; ND[6]
Electrostatic Discharge[22]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
$KClO_3$/dodecane[23]	0/10 @ 1.0[24,25]	0/20 @ 0.125[25]	0/20 @ 0.140[25]	ND[6]
RDX Class 5 Type II[7]	0/10 @ 1.0	0/20 @ 0.0250	0/20 @ 0.095	0/20 @ 0.044
PETN[10]	0/10 @ 1.0	0/20 @ 0.0625	ND[6]	ND[6]

1. DH_{50}, in cm, is by a modified Bruceton method, load for 50% reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.2; 18–20), LANL (21.3–22.7; < 10), IHD (20; 42–46); 3. 180-grit sandpaper data only; 4. Average of two measurements from Table 3; 5. Average of three measurements from Table 3; 6. ND = Not determined; 7. RDX average values from reference 15; 8. 120-grit sandpaper data; 9. 150-grit sandpaper data; 10. From data taken outside of the Proficiency Test; 11. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 12. F_{50}, in kg, is by a modified Bruceton method, load for 50% Reaction; 13. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.2–23.9; 18–19), LANL (22.7–23.6; < 10), IHD (25–35; 41–42); 14. Average of three measurements from Table 5; 15. One value only from Table 5; 16. LLNL and LANL did not perform measurements; 17. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 18. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 19. Measurements performed at 8 fps; 20. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (25; 40–41); 21. Average of three measurements from Table 6; 22. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 23. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3–23.9; 21–22), LANL (22.7–24.0; < 10), IHD (23; 40); 24. LLNL has 510-Ω series resistor in circuit; 25. Average of three measurements from Table 7.

For the current set of ABL friction data, IHD is the only participant that provided any data that can be compared to standards. When comparing with the RDX standard data, $KClO_3$/dodecane mixture appears to be much less sensitive, consistent with the BAM friction results. Currently, there is no ABL friction data for PETN.

Spark sensitivity. Comparing the $KClO_3$/dodecane mixture spark sensitivity values to the corresponding RDX values, the mixture is less spark sensitive than RDX. There are limited values for PETN, but the comparison shows the mixture to be less sensitive.

Thermal sensitivity. The thermal sensitivity of $KClO_3$/dodecane compared to the RDX standard is difficult to assess examining the data in Table 8, because of the lack of exothermic features when using standard DSC sample holders (pin-hole vented). On the surface, this would indicate that the mixture is not thermally sensitive as RDX. However, the data, when using a sealed sample holder, suggest that there may be more chemistry occurring. This is discussed below. At this time, the thermal sensitivity of $KClO_3$/dodecane cannot be reliably assessed by this technique in the standard configuration, so it is not clear that is more or less sensitive than RDX.

4.2 Comparison of results based on participants

There are differences in methodologies and equipment configurations among the participating laboratories, so comparison of results for the same test is useful to highlight any differences in SSST testing methods. Using the average values shown in Table 10, although not statistically rigorous, at least allows for a qualitative comparison of any trends that may be seen among the participants. For impact testing, all participants show about the same sensitivity for the $KClO_3$/dodecane mixture. This is based on data obtained when using the 180-grit sandpaper, and the IDCA participants have agreed to use 180-grit sandpaper all from the same batch for future measurements.

For BAM Friction, LANL average values for both TIL and F_{50} indicate a more sensitive material than the comparable values from LLNL and IHD. This is not the same as seen for RDX, where average values from IHD for F_{50}, show the material more friction sensitive than the other participants. For ESD, LLNL consistently shows a much more stable material, highlighting the large design difference between the LLNL spark testing system and the others. In addition, the ESD averages from IHD indicate a slightly more stable material than the averages from LANL, paralleling the averages for the RDX data from the same laboratories.

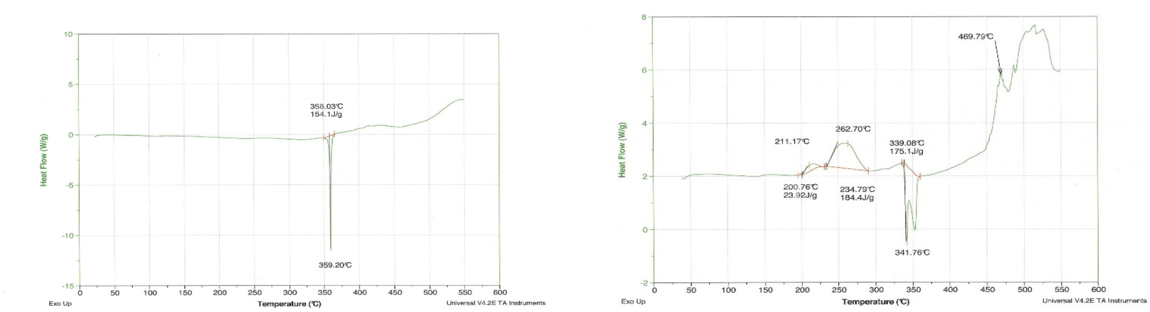

Figure 1. DSC profiles of $KClO_3$/dodecane mixture from pin-hole sample holder (left) and hermetically sealed sample holder (right) at 10°C/min heating rate.

4.3 Behavior of $KClO_3$/dodecane in DSC

As noted in the Results section, the DSC behavior of the $KClO_3$/dodecane mixtures is not well understood. Figure 1 shows the issues observed in performing the DSC of this mixture. The left hand side profile is the mixture being examined using a pinhole sample holder. This sample holder is vented, and typically used for non-volatile samples. Observed is one strong endothermic response that has been assigned to the melting of pure $KClO_3$[3,13,14]. This implies that the other component of the mixture, dodecane, is either completely inert to the $KClO_3$, or is absent. Previous studies on $KClO_3$/fuel by the IDCA and others indicate that it is unlikely that the dodecane is completely inert to $KClO_3$[3,13,14,16]. However, the data in Table 8 shows only endothermic responses in the DSC profiles. Note, there is a very weak exothermic feature in the data from IHD that is unassigned.

Figure 1 right hand side shows the DSC of $KClO_3$/dodecane using a hermitically sealed sample holder. This sample holder is presumed not to leak under moderate pressure. The profile exhibits a substantially different behavior than the profile generated using the pinhole sample holder. Broad exothermic events are visible at

~210°C, ~263°C, ~340°C, and 470°C and above. The high temperature exothermic features, which dominate the profile, have been assigned previously to the decomposition of $KClO_3 \rightarrow KCl + 3/2\ O_2$[14]. The endothermic features at 340 and 350°C might be assigned to the melting of $KClO_3$, but the shape of the feature is not quite what is seen in the pinhole sample holder data. The other exothermic features are not particularly intense and are fairly complicated so full analysis of these is beyond the scope of this report, and will be discussed elsewhere. However, it is important to note that even though the lack of features in data from the pinhole sample holder implies no thermal reactivity, the appearance of exothermic features in the sealed pan, even though weak, implies something energetic is happening in the sample. The application of the standard DSC is just not adequate for evaluating the thermal sensitivity of this sample. Volume and vapor pressure calculations with both sample holders reveals that little if almost no liquid dodecane is in contact with the $KClO_3$ at elevated temperatures, so observing evidence of exothermic reactions is fleeting at best. In previous DSC studies using high pressure sealed sample holders (Setaram), $KClO_3$ mixed with volatile fuels exhibited broad exothermic features in the range of 200 to 250°C just as with this sample. These mixtures have the same volatility-contact issues that $KClO_3$/dodecane has, but the evidence suggests that exothermic features around 200°C in the DSC of the $KClO_3$/dodecane mixture are important in regard to real thermal stability.

4.4 Effect of Sandpaper Grit Size on Impact Data

For the impact test, the method chosen, as listed in Table 2 for all participants, is the Type 12 drop weight test. In this test, the sample is held in place in the anvil with sandpaper. The military specification, MIL-STD-1751A, recommends ("usual usage") but does not require 180-grit sandpaper or garnet paper[17]. As with many explosives safety testing, each laboratory has refined its own criteria for testing, so the choice of the grit size of the sandpaper has been at the discretion of each participant (current configurations)—LLNL, 120-grit; LANL, 150-grit; IHD, 180-grit. Throughout the IDCA testing so far, which includes RDX, $KClO_3$/icing sugar (-100) mixture and $KClO_3$/icing sugar (as received) mixture, the differences in DH_{50} test results have, to some extent, been attributed to the grit size of the sandpaper. These differences have been enough to guide the participants to ultimately decide on using 180-grit sandpaper purchased from one batch and distributed.

The initial impact test results, listed in Table 3, on the mixture of this report, are some of the most varied in the Proficiency test. The LLNL data from using the 120-grit size sandpaper showed the mixture to be much more stable to impact by roughly 4 times compared to the data from the other participants. To understand the origin of this difference, LANL has been performing the impact testing with both 150-grit and 180-grit size sandpaper since the onset of the Proficiency Test and found, generally, the use of the 150-grit paper produced data that indicated the material studied was more stable to impact than the corresponding 180-grit paper data, although the difference was not large. However, the very large difference seen in the 120-grit size produced data in Table 3 prompted LLNL to re-evaluate the impact sensitivity using the 180-grit size sandpaper. The results from the LLNL testing with 180-grit size sandpaper were very similar to the results obtained by LANL and IHD also using the 180-grit size sandpaper, also shown in Table 3.

Characterization of the surface of the sandpapers helps to understand why the differences in the three grit sizes lead to different results. The importance of these reasons has profound implications on what is the appropriate grit size to be using for testing of these solid-solid and solid-liquid mixtures, particularly when the data from the use of 120-grit sandpaper are so dramatically different than the data from the use of the 180-grit sandpaper. Figure 2 shows the scanning electron microscopy (SEM) images of the three sandpapers used in this study. Clearly, there are obvious differences in grain size, spacing and particle density.

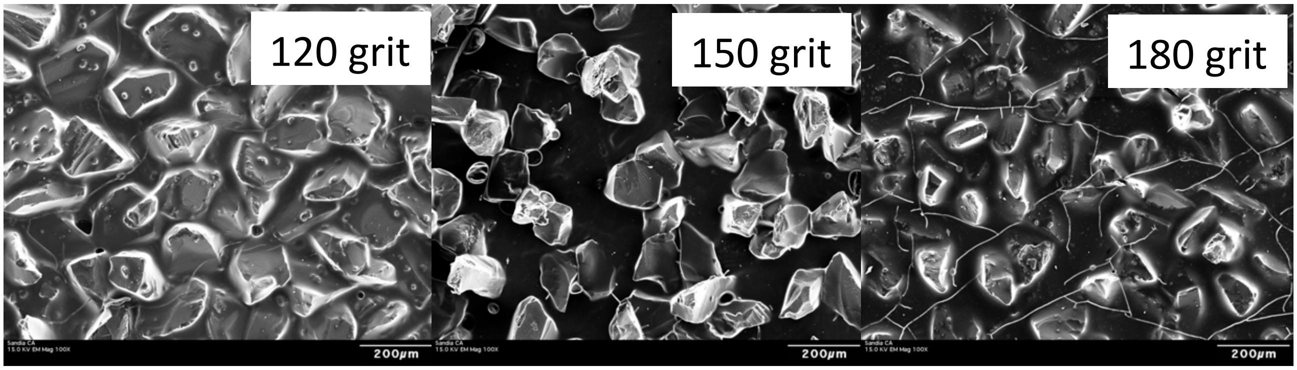

Figure 2. SEM images of sandpaper used in impact testing for $KClO_3$/dodecane

Table 11. Physical properties of selected sandpapers

Property	120-grit	150-grit	180-grit
Particle size (CAMI)[1]	0.115 mm	0.092 mm	0.082 mm
Surface Coverage (from figure[2])	51 particles/mm^2	57 particles/mm^2	54 particles/mm^2
Surface Coverage (Calculated[3])	59 particles/mm^2	115 particles/mm^2	142 particles/mm^2
Volume of particles[4]	0.0765 mm^3	0.0456 mm^3	0.0324 mm^3
Volume of sandpaper[5]	0.115 mm^3	0.092 mm^3	0.082 mm^3
% of total volume[6]	67%	50%	40%

1. From reference 17; 2. Counted manually from Figure 2; 3. Theoretical maximum coverage calculated assuming particles are square with CAMI dimension; 4. From counted number of particles; 5. Assumes 1 mm^2 surface times the particle height; 6. Percentage of volume of the sandpaper that the particles take up.

The particle sizes of U. S. Graded (CAMI standard) sandpaper[18] are shown in Table 11. As expected, the particle size decreases with increasing grit size. The change is about 30% from 120-grit to 180-grit. The surface coverage of particles was also counted manually from the images above. This is a crude method, but the values are surprisingly constant over the surfaces shown here. The theoretical maximum coverage is also listed in Table 11 and is calculated assuming the particles have the same size in every dimension, and represents the maximum number of particles that could fit on the surface assuming identical dimensions and fitting together. This value increases as the grit size increases where the theoretical surface coverage is over twice as much for the 180-grit case compared to the 120-grit case. The volume of the particles also assumes that the single particle is a cube of equal dimensions and the volume is a sum of all the individual volumes using the counted surface coverage particle number. This decreases with increasing grit size. The total volume of the paper assumes that a volume on the surface is defined as a 1 mm x 1 mm x particle size height. This is to represent the area of the sandpaper with a depth the same as the particle for that specific grit size. The % of volume taken by the particles is calculated from the ratio of the total particle volume and the total sandpaper volume. This value decreases as the grit size increases.

There are many theories on what causes a positive reaction in the impact test[19]. One theory is that there must be friction points, pore collapse, or shear points to initiate the reaction. The sandpaper used in these tests certainly provides such a surface for friction or shear points. The amount of points would be the simple explanation, but the values in Table 11 show that the relationship is not that simple. It appears with these sandpapers, the amount of particles remains reasonably constant over the three surfaces. The areas covered as well as the volume accounted for by the particles actually decrease going from the coarsest to the finest. Although this issue will be studied in detail elsewhere, the differences seen in the data obtained from these sandpapers probably is not just the availability of pinch points on the surface, but may also include the nature of the paper itself and/or the adhesive used to hold the grit to the paper. Note that the 120-grit paper is a wet/dry adhesive, while the others are not. In addition, LLNL performed the impact test on 180-grit paper, where the sample was aged over night on the sandpaper. The DH_{50} was found to be 18.5 cm, double of what was found with freshly prepared samples, suggesting that the paper/adhesive may be absorbing or reacting with the dodecane or the dodecane evaporated.

4.5 Comparison with other data

No data appears to be available on $KClO_3$/dodecane mixtures, but some studies have been conducted on $KClO_3$ with carbon-based fuels—Vaseline, motor oil, diesel fuel and nitrobenzene. Generally, impact testing of these mixtures show the mixtures to be more stable than RDX; friction testing show the mixtures to be more sensitive than RDX and sometimes PETN; ESD testing show the mixtures to essentially stable. Because the materials, methods and equipment configurations were not always delineated, the comparison of the data in this table can only be qualitative. The data come from primarily two laboratories—LLNL[20] and LANL[21].

5 CONCLUSIONS

$KClO_3$/dodecane mixture was found through SSST testing to be a moderately sensitive mixture toward impact, friction, and spark handling conditions—generally more sensitive than RDX, but less than PETN (with the impact sensitivity being the exception). Standard thermal testing by DSC probably does not adequately describe the system.

The proficiency study shows that for $KClO_3$/dodecane mixture the current equipment configurations and experimental methods, all participants more or less found the material to have the same impact stability (more sensitive than RDX and PETN). For friction LANL results show the material to have less stability than the corresponding results from IHD and LLNL (less sensitive than PETN). For ESD, LLNL found the material to be insensitive, and IHD and LANL found the material to have the same sensitivity (less than both RDX and PETN). For thermal results, unlike in the case for RDX, where all the participants had results that were virtually identical, and unlike the case of $KClO_3$/icing sugar mixtures, where sampling issues have complicated the interpretation of the results, no prominent exothermic features were seen when using the standard DSC configuration. Sealed sample holder data suggests that there are exothermic events occurring in the 200 to 300°C range.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry

DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

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